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In re Application of:

Takayuki USAMI et al.

Serial No.: 09/966,389

Confirmation No.: 5086

Filed: September 27, 2001

Group Art Unit: 174

Examiner: IP, SIKYIN

For: HIGH-MECHANICAL STRENGTH COPPER ALLOY

DECLARATION UNDER 37 C.F.R. § 1.132

Honorable Commissioner of Patents and Trademarks Washington, D.C. 20231 Sir:

- I, Tatsuhiko EGUCHI, hereby declare and state that:
- 1. I am a Japanese citizen residing at c/o THE FURUKAWA ELECTRIC CO., LTD. of 6-1 Marunouchi 2-chome, Chiyoda-ku, Tokyo, Japan.

I was graduated from Metal Material Department in Engineering Faculty of Tohoku University in March 1987 with a Master's degree and joined The Furukawa Electric Co., Ltd. in April 1987. From 1987 to 1997, I was engaged in research and development of copper alloys for electric and electronic machinery and tools, in the Metal Research Center of the said company. From 1997 to 2001, I was engaged in research and development of manufacturing technique of copper alloys for electric and electronic machinery and tools, in Technical and Engineering Department in Metals Group of the said company. Since

2001, I have been engaged in research and development of copper alloys for electric and electronic machinery and tools, in the Metal Research Center.

I am intimately familiar with the contents of United States Patent Application Serial No. 09/966,389, filed on September 27, 2001, its prosecution before the United States Patent & Trademark Office, and the references cited therein.

- 2. I have studied the contents of the cited unexamined published Japanese patent application No. 11-43731, and unexamined published Japanese patent application No. 6-41660.
- 3. To show the superiority of the present invention, the following tests were conducted, by me or under my supervision:

Test

- (1) The alloy of the present invention and those of JP-A-6-41660 (JP'660) and JP-A-11-43731 (JP'731) were compared in the following manner.
- 1) Preparation of the samples

Copper alloy 101a having the same alloy composition as Example 10 in JP'660, was prepared. Further, Copper alloy 102a having the same alloy composition as Example 3 in JP'731, was prepared.

Further, Copper alloy A as described in the present specification was also reproduced.

The compositions of the alloys 101a and 102a were shown below Tables I-1 and I-2. These alloys were reproductions of

those described in the cited references according to the description described in the cited references within the claimed range of the references, though the contents of the elements in those alloys were slightly different from those described in the cited references. It was difficult to prepare the exactly same alloys as described in JP'660 and JP'731. For reference, the compositions described in JP'660 and JP'731 are excerpted and also shown in Tables I-1 and I-2.

Table I-1 mass%

l lula l		Ni	Si	Sn	Mg	Zn	Fe	Мо	Cu
101a (Example 10	Described content	1.95	0.48	0.52	0.19	0.95	0.05	0.0008	Balance*1
in JP'660)	Obtained content	1.95	0.45	0.50	0.18	1.01	0.06	0.0009	Balance ¹¹

*1 With unavoidable impurities

Table I-2 mass%

Alloy No.		Ni	Si	Sn	Mg	Zn	S	С	Cu
102a (Example 3	Described content	3.2	0.7	0.5	0.1	1.0	0.0015	0.003	Balance ^{*1}
in JP'731)	Obtained content	3.17	0.60	0.49	0.09	1.08	0.001	0.002	Balance*1

*1 With unavoidable impurities

Further, these alloys were worked with the conditions as shown in the Tables II-1, and II-2 below. That is, Alloy101a was worked in the same manner as in JP'660 to obtain Sample 101, while Alloy 102a was worked in the same manner as in JP'731 to obtain Sample 102.

Further, the following copper alloy 201a was worked in the same manner as Sample 1 (Alloy A) in the present specification, to manufacture Sample 201. The compositions of the alloys A and 201a are shown in Table I-3 below.

Table I-3

mass%

Alloy No.	Ni	Si	Mg	Şn	Zn	S	Cu
Α	3.9	0.90	0.10	0.18	0.49	0.002	Balance*1
201 a	3.8	0.94	0.15	0.50	0.09	0.002	Balance ¹

*1 With unavoidable impurities

Particulars for the working conditions are shown in the following Tables II-1, II-2 and II-3.

In the Tables II-1 and II-2, when the working conditions utilized for Samples 101 and 102 in this test corresponded to those described in JP'660 and JP'731, it is designated as "O", while when the conditions did not corresponded to, it is designated as "x".

Table II-1 Manufacturing conditions for Sample 101

		Cone	ditions for Sample	101
Steps	Conditions described in JP'660	Conditions	Correspondence with the described condition	Remarks
Ingot obtained by melting and casting	(mmxmmxmm)	35×100×180 (mmxmmxmm)	×	A small-size mold was used.
Re-heating	750∼980 °C	850 °C	0	Heated for 1hr
Hot-rolling	11t	12.5t	×	Because of a limitation in laboratory equipment, accuracy of thickness was rather inferior.
Water cooling		Water cooling	0	-
Chamfering	0.5mm each for both the upper and lower sides.	2.5mm in total from both the upper and lower sides.	×	The thickness after chamfering was adjusted so as to be the same thickness as described in JP'660.
Cold-rolling	1.32t (87%)	1.32t	0	-
Process annealing	400∼650 °C × 1hr	550°C × 1hr	0	-
Cold-rolling	0.33t (75%)	75%	0	-
Solution heat treatment	750∼950 °C × 5∼300 sec	750 × 30sec	0	-
Rapid cooling	750~500 °C: 40°C/sec	100 °C/sec or more	0	-
Precipitation treatment (Aging)	400∼500 °C × 3hr	475 °C × 3hr	0	-
Cold-rolling	0.247t (25%)	25%	0	-
Stress relief annealing (low- temperature annealing)	250∼350 °C × 1hr	300 °C × 1hr	0	-

Table II-2 Manufacturing conditions for Sample 102

		T		
		Co	nditions for Samp	le 102
Steps	Conditions described in JP'731	Conditions	Correspondence with the described condition	Remarks
Ingot obtained by melting and casting	50 × 80 × 200 (mmxmmxmm)	35 × 100 × 180 (mmxmmxmm)	×	A small-size mold was used.
Re-heating	930 °C	930 °C	0	Heated for 1hr
Hot-rolling	15t	16.7t	×	Because of a limitation in laboratory equipment, accuracy of thickness was rather inferior.
Water cooling		Water cooling	0	
Chamfering	Grinder (No particular thickness was mentioned.)	2.7mm in total from both the upper and lower sides.	×	The thickness after chamfering was adjusted so as to be the same thickness as described in JP'731.
Cold-rolling	0.36t	0.36t	0	_
Solution heat treatment	650~850 °C x 20sec	800°C × 20sec	0	
Rapid cooling			0	-
Cold-rolling	0.25t	0.25t	0	
Precipitation treatment (aging)	450~500 °C x 2hr	475 °C × 2hr	0	-

Table II-3 Manufacturing conditions for Samples 1 and 201

Steps	Conditions for Sample 1 as described in the present specification	Conditions for Sample 201
Ingot obtained by melting	$30 \times 100 \times 150$	30 × 100 × 200
and casting	(mmxmmxmm)	(mmxmmxmm)
Re-heating	1000 °C × 30min	1000 °C × 10min
. Hot-rolling	12t	12t
Rapid cooling		Water cooling
Chamfering	1.5 mm from both the	0.5 mm from both the
<u> </u>	upper and lower sides	upper and lower sides.
Cold-rolling	0.28t	0.28t
Solution heat treatment	875∼900 °C x 15sec	875 °C x 15sec
Rapid cooling	15°C/sec or more	200°C/sec or more
Precipitation treatment (aging)	475 °C × 2hr	475 °C × 2hr
Cold-rolling	0.25t	0.25t
Low-temperature annealing	350 °C × 2hr	350 °C × 2hr

2) Evaluation of the samples

These samples were evaluated on the following characteristics.

2-1) Physical properties

Table III-1

No.	TS (MPa)	El (%)	EC (%)	Remarks
Sample 101 (Example 10 in JP'660)	740 (815)	6.0 (8)	36.4 (38)	Comparative example
Sample 102 ¹⁾ (Example 3 in JP'731)	758 (900)	10.5	36.7 (40)	Comparative example
Sample 201	928	7.6	34.0	This invention
Sample 1	880	12	33	This invention

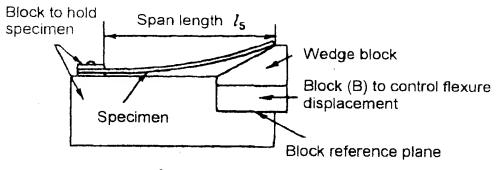
Note:

- 1) Since the manufacturing steps disclosed in JP'731 did not include a low-temperature annealing step, the values shown for this sample were ones obtained with the sample after aging, different from other samples.
- 2) The values in the parenthesis were those shown in the cited references.

2-2) Stress Relaxation Property

(a) The samples were evaluated, by applying the stress load of 600 MPa.

The drawing shown below shows an apparatus used to evaluate the stress relaxation property.



$$\delta_o = \frac{\sigma_0 \, ls^2}{1.5 \, Eh}$$

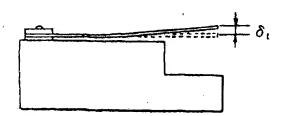
 δ_0 = Initial flexure amount (mm)

 σ_0 = Initial bending stress (N/mm^2)

 $I_s = \text{Span length } (mm)$

E = Flexure coefficient (kN/mm²)

h =Plate thickness or wire diameter (mm)



 δ_t = Permanent flexure amount after a prescribed period (mm)

The results obtained are shown below. (The samples were maintained in a constant temperature chamber at 150 $^{\circ}\text{C}\text{)}.$

Table III-2

No.	Stress relaxation ratio with stress load of 600MPa (%) *1									
	25h	50h	100h	200h	400h	600h	800h	1000h		
Sample 101 (Example 10 in JP'660)	4.8 (65%)	6.6 (55%)	9.1 (55%)	11.5 (50%)	13.4 (49%)	15.0 (47%)	16.1 (47%)	17.1 (46%)		
Sample 102 (Example 3 in JP'731)	5.9 (53%)	7.5 (48%)	9.4 (53%)	12.1 (47%)	14.2 (46%)	15.1 (47%)	16.0 (47%)	16.7 (47%)		
Sample 201	3.1	3.6	5.0	5.7	6.6	7.1	7.5	7.9		
Sample 1	ND	ND	ND	ND	ND	ND	ND	8		

Note: *1 The values in parenthesis represent the relaxation amount obtained by the following equation: (Ratio of Sample 201)/(Ratio of Sample 101 or 102)x100

The smaller the stress relaxation ratio (S.R.R.) is, the better the stress relaxation property for the alloy is. On the other hand, the larger the stress relaxation amount (shown in parenthesis, as a relative value) is, the better the stress relaxation property is. The stress relaxation property is judged to be good when the stress relaxation ratio (S.R.R.) was 10% or less, and it is judged to be poor when the S.R.R. was more than 10%. This standard is decided based on the fact that a conventionally utilized beryllium-Cu alloy JIS C 1753 has the stress relaxation property of 10%. Therefore, if a sample exhibits less than 10% of S.R.R., this sample has a superior stress relaxation property compared to a conventionally used Be-Cu alloy.

In this connection, each of the working examples of the present invention demonstrated in the present specification exhibited the stress relaxation ratio of 10% or less, even after maintained in a constant temperature chamber at 150 °C for 1,000 hours.

(2) Data to show the criticality of the range of a/b

The following results were obtained according to the test method conducted in the same manner as in working examples in the present specification.

Table IV

ing ate						
Peeling of plate	0	0	0	0	0	0
Electric conductivity %IACS	32	32	33	33	33	33
Bending property (presence Elongation or absence of cracks)	80	6	10	12	1	=
	×	0	0	0	0	0
SRR %	25	19	6	&	6	10
Tensile strength N/mm²	920	910	895	875	875	885
Shape characteristics of crystal grain (a/b)	2.0	1.7	1.5	1.0	0.8	0.7
Crystal grain diameter (mm)	0.005	0.005	0.020	0.005	0.005	0.005
Sample No.	20	19	41	42	43	. 5
Ingot No.	∢ .	А	٧	∢	∢	∢
Classification Ingot Sample No. No.	Comparative example	Comparative example	This invention	This invention	This invention	This invention

designated as "x". When the peeling property of plate was good it is designated as "O", while when the peeling property Note: When the bending property was good it is designated as "O", while when the bending property was poor it is was poor it is designated as "x". The data already of record in the specification and the supplemental data submitted herewith demonstrate unexpectedly superior results of the claimed high-mechanical strength copper alloy over those of the cited prior art.

4. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date: August 9 , 2002

Vatsuhileo Equeli Tatsuhiko EGUCHI